Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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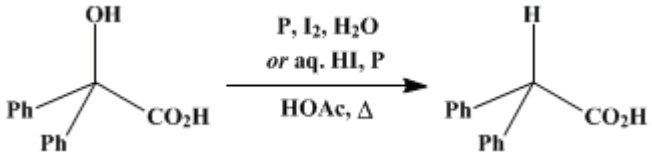
DIPHENYLACETIC ACID

[Acetic acid, diphenyl-]



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1. Procedure

In a 1-l. round-bottomed flask are placed 250 cc. of glacial acetic acid, 15 g. of red phosphorus, and 5 g. of iodine. The mixture is allowed to stand for fifteen to twenty minutes until the iodine has reacted, and then 5 cc. of water (Note 1) and 100 g. (0.44 mole) of benzilic acid (p. 89) are added. A reflux condenser is attached, and the mixture is boiled continuously for at least two and one-half hours. After the reaction is complete, the hot mixture is filtered with suction to remove the excess red phosphorus (Note 2). The hot filtrate is slowly poured into a cold, well-stirred, filtered solution of 20–25 g. of sodium bisulfite in 1 l. of water (Note 3). This procedure removes the excess iodine and precipitates the diphenylacetic acid as a fine white or slightly yellow powder (Note 4). The product is filtered with suction, washed with cold water, and dried thoroughly on filter paper. The yield is 88–90 g. (94–97 per cent of the theoretical amount) of a solid melting at 141–144° (Note 5). If a crystalline product is desired, the acid is dissolved in about 500 cc. of hot 50 per cent alcohol and then cooled. The melting point after recrystallization is 144–145°.

2. Notes

1. In place of phosphorus, iodine, and water, a dilute solution of hydriodic acid and phosphorus may be used.
2. If difficulties are encountered in filtering the hot acetic acid solution through filter paper, an asbestos filter may be prepared and used to advantage.
3. Some samples of sodium bisulfite caused part of the diphenyl-acetic acid to dissolve. This can always be avoided if, after the solution of sodium bisulfite is prepared, a current of sulfur dioxide is passed in, until the solution is acid to litmus.
4. Sometimes, if the acetic acid solution is poured into the water too rapidly, the product will be slightly pink and a reprecipitation from acetic acid solution will be necessary.
5. The melting points of benzilic acid and diphenylacetic acid lie very close together. However, it is very easy to test for complete reduction by treating a little of the product with cold concentrated sulfuric acid. If even a trace of benzilic acid remains the sulfuric acid will turn red.

3. Discussion

Diphenylacetic acid can be prepared by the reduction of benzilic acid with hydriodic acid and red phosphorus,[1] by carbonation of the reaction product of phenylsodium and diphenylmethane,[2] and by hydrolysis of 1,1-dichloro-2,2-diphenylethylene.[3]

References and Notes

1. Jena, Ann. **155**, 84 (1870); Klingemann, Ann. **275**, 84 (1893); Zinssen, Ber. **24**, 3556 (1891); Chichibabin, Ber. **44**, 442 (1911).
2. I. G. Farbenind. A.-G., Ger. pat. 671, 098 [C. A. **33**, 3391 (1939)].

3. Sheibley and Prutton, J. Am. Chem. Soc. **62**, 840 (1940).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

red phosphorus

alcohol (64-17-5)

sulfuric acid (7664-93-9)

acetic acid (64-19-7)

sulfur dioxide (7446-09-5)

PHOSPHORUS (7723-14-0)

sodium bisulfite (7631-90-5)

iodine (7553-56-2)

Benzilic acid (76-93-7)

Diphenylmethane (101-81-5)

hydriodic acid (10034-85-2)

Diphenylacetic acid,
Acetic acid, diphenyl-,
diphenyl-acetic acid (117-34-0)

phenylsodium

1,1-dichloro-2,2-diphenylethylene